U. S. Gov't
Master
Specification
No. 4a

DEPARTMENT OF COMMERCE
BUREAU OF STANDARDS
George K. Burgess, Director

CIRCULAR OF THE BUREAU OF STANDARDS, No. 82

[3d Edition. Issued April 26, 1927]

UNITED STATES GOVERNMENT MASTER SPECIFICATION FOR OIL, LINSEED, RAW [1]

FEDERAL SPECIFICATIONS BOARD SPECIFICATION No. 4a

[Revised March 5, 1927]

This specification was officially promulgated by the Federal Specifications Board on February 3, 1922, for the use of the departments and independent establishments of the Government in the purchase of raw linseed oil.

[The latest date on which the technical requirements of this revision of this specification shall become mandatory for all departments and independent establishments of the Government is June 6, 1927. They may be put into effect, however, at any earlier date.]

CONTENTS

		Page
I.	General specifications	1
II.	Types	1
III.	Material and workmanship	2
IV.	General requirements	2
V.	Detail requirements	2
VI.	Methods of sampling, testing, and basis of purchase	2
	1. Sampling	2
	2. Testing	3
	3. Reagents	6
	4. Basis of purchase	8
VII.	Packing	8
VIII.	Notes	8

I. GENERAL SPECIFICATIONS

There are no general specifications applicable to this specification.

II. TYPES

This specification covers two types of raw linseed oil: A, normal iodine number; B, high iodine number.

[1] For boiled linseed oil see United States Government Master Specification for Boiled Linseed Oil, Federal Specifications Board specification No. 475a. (B. S. Circular No. 330.)

43256°—27

III. MATERIAL AND WORKMANSHIP

See detail requirements.

IV. GENERAL REQUIREMENTS

See detail requirements.

V. DETAIL REQUIREMENTS

Raw linseed oil shall be pure oil expressed from flaxseed, and shall conform to the following requirements:

	Maximum	Minimum
Foots:		
Heated oil, per cent by volume	1.0	
Chilled oil, do	4.0	
Specific gravity 15.5/15.5° C	.935	0.9300
Acid number	5.5	
Saponification number	195.0	189.0
Unsaponifiable matter, per cent	1.50	
Iodine number [1], Wijs		175.0
Loss on heating at 105 to 110° C, per cent	.2	
Color	Not darker than a freshly prepared solution of 1.0 g potassium bichromate in 100 cc pure concentrated sulphuric acid (sp. gr. 1.84).	

[1] When high iodine number type of raw linseed oil is specified by the purchaser, the iodine number must be not less than 188 and the oil shall conform to all of the other requirements.

VI. METHODS OF SAMPLING, TESTING, AND BASIS OF PURCHASE

Deliveries will, in general, be sampled and tested by the following methods, but the purchaser reserves the right to use any additional available information to ascertain whether the material meets the specification.

1. SAMPLING

The method of sampling given under (*a*) below should be used whenever it is feasible to apply it. To meet conditions when (*a*) is not applicable, method (*b*), (*c*), or (*d*) is to be used according to the special conditions that obtain.

(*a*) During Loading of Tank Cars or Filling of Containers for Shipment at the Factory.—The purchaser's inspector shall draw a sample at the discharge pipe where it enters the receiving vessel or vessels. The total sample shall be not less than 5 gallons and shall be a composite of small samples of not more than 1 pint each, taken at regular intervals during the entire period of loading or filling.

The sample thus obtained shall be thoroughly mixed, and from this composite sample three portions of not less than 1 quart each shall be placed in clean, dry glass bottles or tin cans which must be

filled with the sample and securely stoppered with new clean corks or well-fitting metal covers or caps. These shall be sealed and labeled distinctly by the inspector, and one delivered to the buyer, one to the seller, and the third held for check in case of dispute.

(*b*) From Loaded Tank Cars or Other Large Vessels.—The total sample shall be not less than 5 gallons and shall be a composite of numerous small samples of not more than 1 pint each, taken from the top, bottom, and intermediate points by means of a glass or metal container with removable stopper or top. This device attached to a suitable pole is lowered to the various desired depths when the stopper or top is removed and the container allowed to fill. The sample thus obtained is handled as in (*a*).

(*c*) Barrels and Drums.—Not less than 5 per cent of the packages in any shipment or delivery of barrels and drums shall be sampled. The packages shall be shaken, rolled, and stirred to mix the contents thoroughly. The samples from the individual containers shall be taken through the bunghole or holes not less than 1¼ inches in diameter bored in the head or side for the purpose. The apparatus for drawing the sample shall consist of a glass tube about 1 inch in diameter and somewhat longer than the length or diameter of the oil container, a conical stopper that will fit the glass tube and is not more than ½ inch long fastened to a stiff metal rod not more than ¼ inch in diameter and not less than 4 inches longer than the glass tube. The stopper is lowered by the rod until it rests on the bottom of the cask, the tube slipped down slowly over the rod, and finally pressed on the stopper. By holding tube and rod, the column of oil can then be removed. This process is repeated until the required amount of samples is obtained, which shall be not less than 2 gallons. This is mixed and handled as in (*a*).

(*d*) Small Containers, Cans, Etc., of 10 Gallons or Less.—Small containers, cans, etc., of 10 gallons or less should be sampled while filling by method (*a*) whenever possible. When method (*a*) is not applicable, it is mutually agreed that: In all cases the total sample taken shall not be less than 3 quarts. This shall be obtained by taking at least one package from each lot of not more than 300 packages. The sample thus taken shall be thoroughly mixed and subdivided as in (*a*).

2. TESTING

All tests shall be made on oil that has been thoroughly agitated before removal of a portion for analysis.

(*a*) Foots.—*General test.*—With all materials at a temperature between 20 and 27° C., mix, by shaking for exactly one minute in a graduated tube, 25 cc of the well-shaken sample of oil, 25 cc of acetone, and 10 cc of acid calcium chloride solution. Then clamp the tube in an upright position and allow to settle for 24 hours.

The temperature during this period should be between 20 and 27° C. The graduated tube shall be of not less than 70 cc capacity and shall have at least 50 cc graduated in 0.1 cc. The diameter of the tube shall be such that the 50 cc graduated portion shall be not less than 40 cm or more than 60 cm in length. The volume of the stratum lying between the clear calcium chloride solution and the clear acetone and oil mixture is read to 0.1 cc or fraction thereof. This reading multiplied by four expresses the amounts of foots present as percentage by volume of the oil taken.

Heated oil.—Heat a portion of the oil to 65° C., hold it within 2° C. of that temperature for 10 minutes, then cool it to room temperature (20 to 27° C.). Promptly make the general foots test as described above.

Chilled oil.—Heat a portion of the oil to 65° C., hold it within 2° C. of that temperature for 10 minutes, then place it in a clean, dry bottle, stopper tightly, and place in a cracked ice and water mixture (0° C.) for exactly two hours. At the end of this time place the bottle in a large bath of water at 25° C. and keep it there for 30 minutes, then promptly make the general foots test as described above.

(*b*) Specific Gravity.—Determine at 15.5/15.5° C. by any convenient method that is accurate within two points in the fourth decimal place.

(*c*) Acid Number.—Weigh from 5 to 10 g of the oil. Transfer to a 300 cc Erlenmeyer flask. Add 50 cc of a mixture of equal parts by volume of 95 per cent ethyl alcohol and c. p. reagent benzol. (This mixture should be previously titrated to a very faint pink with dilute alkali solution, using phenolphthalein as an indicator.) Add phenolphthalein indicator and titrate at once to a faint permanent pink color with standard sodium or potassium hydroxide solution. Calculate the acid number (milligrams KOH) per gram of oil.

(*d*) Saponification Number.—Weigh about 2 g of the oil and transfer to a 300 cc Erlenmeyer flask. Add 25 cc of alcoholic sodium hydroxide or potassium hydroxide solution. Put a condenser loop inside the neck of the flask and heat on a steam bath for one hour. Cool, add phenolphthalein as indicator, and titrate with 0.5 N H_2SO_4. Run two blanks with the alcoholic alkali solution. These should check within 0.1 cc 0.5 N H_2SO_4. From the difference between the number of cubic centimeters of 0.5 N H_2SO_4 required for the blank and for the determination, calculate the saponification number (milligrams KOH required for 1 g of the oil).

(*e*) Unsaponifiable Matter.—Weigh 8 to 10 g of the oil and transfer to a 250 cc long-neck flask. Add 5 cc of a concentrated solution of sodium hydroxide (equal weights of NaOH and H_2O)

and 50 cc of 95 per cent ethyl alcohol. Put a condenser loop inside the neck of the flask and boil for two hours. Occasionally agitate the flask to break up the liquid, but do not project the liquid onto the sides of the flask. At the end of two hours remove the condenser and allow the liquid to boil down to about 25 cc.

Transfer to a 500 cc glass-stoppered separatory funnel, rinsing with water. Dilute with water to 250 cc, add 100 cc of redistilled ether. Stopper and shake for one minute. Let stand until the two layers separate sharp and clear. Draw all but one or two drops of the aqueous layer into a second 500 cc separatory funnel and repeat the process, using 60 cc of ether. After thorough separation draw off the aqueous solution into a 400 cc beaker, then the ether solution into the first separatory funnel, rinsing down with a little water. Return the aqueous solution to the second separatory funnel and shake out again with 60 cc of ether in a similar manner, finally drawing the aqueous solution into the beaker and rinsing the ether into the first separatory funnel.

Shake the combined ether solution with the combined water rinsings and let the layers separate sharp and clear. Draw off the water and add it to the main aqueous solution. Shake the ether solution with two portions of water (about 25 cc each). Add these to the main water solution.

Swirl the separatory funnel so as to bring the last drops of water down to the stopcock and draw off until the ether solution just fills the bore of the stopcock. Wipe out the stem of the separatory funnel with a bit of cotton on a wire. Draw the ether solution (portion wise if necessary) into a 250 cc flask and distill off. While still hot, drain the flask into a small weighed beaker, rinsing with a little ether. Evaporate this ether, cool the beaker, and weigh. (The unsaponifiable oil from adulterated drying oils may be volatile and as a consequence may evaporate on long heating. Therefore, heat the beaker on a warm plate, occasionally blowing out with a current of dry air. Discontinue heating as soon as the odor of the ether is gone.)

(*f*) Iodine Number.—Place a small quantity of the sample in a small weighing burette or beaker. Weigh accurately. Transfer by dropping from 0.09 to 0.15 g of oil to a 500 cc bottle, having a well-ground glass stopper, or an Erlenmeyer flask, having a specially flanged neck for the iodine tests. Reweigh the burette or beaker and determine the amount of the sample used. Add 10 cc of chloroform. Whirl the bottle to dissolve the sample. Add 10 cc of chloroform to each of two empty bottles or flasks like that used for the sample. Add to each bottle 25 cc of the Wijs solution and let stand with occasional shaking for one hour in a dark place at a temperature of from 21 to 23° C. Add 10 cc of the 15 per cent potassium iodide

solution and 100 cc of water. Titrate with 0.1 *N* sodium thiosulphate, using starch as an indicator. The titrations on the two blank tests should agree within 0.1 cc. From the difference between the average of the blank titrations and the titration on the samples, and the iodine value of the thiosulphate solution, calculate the iodine number of the samples tested. (Iodine number is given in centigrams of the iodine to 1 g of sample.)

(*g*) Loss on Heating at 105 to 110° C.—Place 10 g of the oil in an accurately weighed 50 cc Erlenmeyer flask and weigh. Heat in an oven at a temperature between 105 and 110° C. for 30 minutes, then cool and weigh. Calculate the percentage loss. This determination shall be made in a current of carbon dioxide.

(*h*) Color.—Prepare a fresh solution of 1 g pure potassium bichromate in 100 cc of pure concentrated colorless sulphuric acid (specific gravity 1.84). Place the oil and color solution in separate thin-walled clear glass tubes of the same diameter (1 to 2 cm) to a depth of not less than 25 cm and compare the depths of color by looking transversely through the columns of liquid by transmitted light.

3. REAGENTS

(*a*) Acetone.—Acetone that will pass the specification of the United States Pharmacopoeia.

(*b*) Acid Calcium Chloride Solution.—Saturate with calcium chloride a mixture of 90 parts water and 10 parts concentrated hydrochloric acid (specific gravity 1.2).

(*c*) Standard Sodium Hydroxide Solution.—Prepare a stock concentrated solution of sodium hydroxide by dissolving hydroxide in water in the proportion of 200 g NaOH to 200 cc of water. Allow this solution to cool and settle in a stoppered bottle for several days. Decant the clear liquid from the precipitate of sodium carbonate into another clean bottle. Add clear barium hydroxide solution until no further precipitate forms. Again allow to settle until clear. Draw off about 175 cc and dilute to 10 liters with freshly boiled distilled water. Preserve in a stock bottle provided with a large guard tube filled with soda lime. Determine the exact strength by titrating against pure benzoic acid (C_6H_5COOH), using phenolphthalein as indicator. (See Bureau of Standards Scientific Paper No. 183.) This solution will be approximately one-fourth normal, but do not attempt to adjust it to any exact value. Determine its exact strength and make proper corrections in using it.

(*d*) Alcoholic Soduim Hydroxide Solution.—Dissolve pure sodium hydroxide in pure 95 per cent ethyl alcohol in the proportion of about 22 g per 1,000 cc. Let stand in a stoppered bottle. Decant

the clear liquid into another bottle and keep well stoppered. This solution should be colorless or only slightly yellow when used.

(*e*) STANDARD SODIUM THIOSULPHATE SOLUTION.—Dissolve pure sodium thiosulphate in distilled water that has been well boiled to free it from carbon dioxide in the proportion so that 24.83 g crystallized sodium thiosulphate will be present in 1,000 cc of the solution. It is best to let this solution stand for about two weeks before standardizing. Standardize with pure resublimed iodine. (See Analytical Chemistry, Treadwell-Hall, vol. 2, 6th ed., p. 551.) This solution will be approximately decinormal, and it is best to leave it as it is after determining its exact iodine value, rather than to attempt to adjust it to exactly decinormal strength. Preserve in a stock bottle provided with a guard tube filled with soda lime.

(*f*) STARCH SOLUTION.—Stir up 2 to 3 g of potato starch or 5 g soluble starch with 100 cc of 1 per cent salicylic acid solution, add 300 to 400 cc boiling water, and boil the mixture until the starch appears to be dissolved. Dilute to 1 liter.

(*g*) POTASSIUM IODIDE SOLUTION.—Dissolve 150 g of potassium iodide free from iodate in distilled water and dilute to 1,000 cc.

(*h*) WIJS SOLUTION.—The preparation of the iodine monochloride solution presents no great difficulty, but it should be done with care and accuracy in order to obtain satisfactory results. There shall be in the solution no sensible excess either of iodine or more particularly of chlorine over that required to form the monochloride. This condition is most satisfactorily attained by dissolving in the whole of the acetic acid to be used the requisite quantity of iodine, using a gentle heat to assist the solution, if it is found necessary. Dissolve iodine in glacial acetic acid that has a melting point of 14.7 to 15° C. and is free from reducing impurities in the proportion so that 13 g of iodine will be present in 1,000 cc of solution. Set aside a small portion of this solution while pure and pass dry chlorine into the remainder until the halogen content of the solution is doubled. Ordinarily it will be found that by passing the chlorine into the main part of the solution until the characteristic color of free iodine has just been discharged, there will be a slight excess of chlorine which is corrected by the addition of the requisite amount of the unchlorinated portion until all free chlorine has been destroyed. A slight excess of iodine does little or no harm, but excess of chlorine must be avoided.

(*i*) HALF NORMAL SULPHURIC ACID SOLUTION.—Add about 15 cc of sulphuric acid (1.84 specific gravity) to distilled water, cool and dilute to 1,000 cc. Determine the exact strength by titrating against freshly standardized sodium hydroxide or by any other accurate method. Either adjust to exactly half normal strength or leave as originally made, applying appropriate correction.

4. BASIS OF PURCHASE

Material is to be purchased by weight or volume, as specified in the contract. When purchased by volume, 1 gallon of oil shall mean 231 cubic inches at 15.5° C.

VII. PACKING

Packing shall be in accordance with commercial practice unless otherwise specified.

VIII. NOTES

This specification supersedes that part of Federal Specifications Board specification No. 4 (B. S. Circular No. 82, 2d ed.) which covered raw linseed oil. For specification for boiled linseed oil, see Federal Specifications Board specification No. 475a (B. S. Circular No. 330). The specification for refined linseed oil contained in Federal Specifications Board specification No. 4 (B. S. Circular No. 82, 2d ed.) is revoked.

For formulas and methods of using this material and information regarding the use of other specification paint materials, see Bureau of Standards Technologic Paper No. 274, entitled "Use of United States Government Specification Paints and Paint Materials."

ADDITIONAL COPIES
OF THIS PUBLICATION MAY BE PROCURED FROM
THE SUPERINTENDENT OF DOCUMENTS
GOVERNMENT PRINTING OFFICE
WASHINGTON, D. C.
AT
5 CENTS PER COPY